

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of  
Maria Anna Wubben et al.

Serial No. 08/776,321

Examiner: C.E. Sherrer

Filed: April 15, 1997

Art Unit: 1761

For: PECTINS AS FOAM STABILIZERS FOR BEVERAGES HAVING A  
FOAM HEAD

DECLARATION of Alexandra J.M. Wijsman

Alexandra Johanna Mathilda Wijsman herewith declares as follows:

1. I presently reside at Hondsdrafveld 23, 3448 EC Woerden, the Netherlands.

2. I am an employee of Heineken Technical Services B.V. at Zoeterwoude, the Netherlands, the assignee of the present invention. I am a graduate of the Agricultural University of Wageningen, with a specialization in food technology. Since 1995 I have been involved in research on raw materials for the production of beer, such as polysaccharides.

3. In order to determine the effect of the amount of pectin on foam stability of beer, an experiment was conducted using different amounts of hop-pectin. I conducted part of the experiment; the other parts were conducted in my presence. The experiments and results are described as follows.

Pectin preparations (extract from CO<sub>2</sub> extract residues) were obtained by an extraction procedure using the same principles as described in the patent application on page 14, however, modified to take into account industrial size unit operations.

The extract contained 42.9 % w/w of pectin (= anhydrogalacturonic acid), the remainder being mainly sugars and minerals, and a small amount of denatured (insoluble) protein (8% w/w) and phenolic compounds (2% w/w).

4. Hop pectin preparations were dissolved in water before being added to reference beer (pilsner beer) in amounts of 1, 5, 10, 15, 20, 25 and 30 g pectin per hectolitre of beer (based on actual pectin (anhydrogalacturonic acid, AUA) content). For the amounts of 1, 5 and 10 g/hl the pectin preparations were dissolved in 5 ml of water and the other amounts were dissolved in 10 ml of water. The bottles were shaken for 48 hours at room temperature. The foam stability was then determined using the Nibem foam meter.

5. The results of the test were as follows.

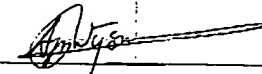
Foam stability of beer after addition of several quantities of hop pectin  
AUA content hop pectin: 42.9% w/w

Samples	Pectin addition g pectin preparation/ hl	Pectin addition as AUA g AUA / hl	Foam stability (sec)			Foam improvement (sec)
					average	
Reference	-	-	289	285	287	-
Hop pectin	2.3	1	327	324	326	39
Hop pectin	11.7	5	357	354	356	69
Hop pectin	23.3	10	380	382	381	94
Hop pectin	35.0	15	456	422	439	152
Hop pectin	46.6	20	480	410	445	158
Hop pectin	58.3	25	499	479	489	202
Hop pectin	69.9	30	473	477	475	188

6. The undersigned declares that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that wilful false statements so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the Unites States Code, and that such wilful false statements so made may jeopardize the validity of the document, or application, or any patent issuing thereon.

Signed this 29 day of October, 1999

By



Alexandra J.M. Wijsman

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HEINEKEN HTS R&amp;D

NO.414

P.2/5

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Examiner: C.E. Sherrer

Art Unit: 1761

D E C L A R A T I O N of Albert Doderer

Albert Doderer herewith declares as follows:

1. I presently reside at Albast 47, 2719 TV  
Zoetermaer, the Netherlands.

2. I am an employee of Heineken Technical  
Services B.V. at Zoeterwoude, the Netherlands, the assignee  
of the present invention. I am a graduate of the Free  
University Amsterdam with a specialisation in  
biology/biochemistry. Since 1985 I have been involved in  
research on raw materials for the production of beer. I am a  
co-inventor of the present invention.

3. In the period the present invention was made,  
prior to August 1994, various experiments were conducted with  
hop pectins extracted from several hop varieties and several  
parts of hop plants. In addition, the influence of the amount  
of hop pectin on beer was investigated.

Pectin preparations (extracts) were obtained by the  
extraction procedure described in the patent application on  
page 14. The hop pectin preparations, which were added, were  
not 100% pectin. Rather, the preparations were approximately  
55-75% pectin (as anhydrogalacturonic acid); see page 17,  
Annex 6, of the patent application.

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Hop pectin preparations were dissolved in 5 ml water before being added to reference beer (pilsner beer) in amounts which resulted in the addition of approximately 3 to 7 grams of pectin per hectolitre of beer. The bottles were shaken for 60 hours at room temperature. The foam stability was then determined using the Nibem foam meter.

The experiments were done either by me, or under my direct supervision.

The results of these experiments were included in the above-referenced patent application, and a summary of the most relevant ones is given in the following table (see the table below).

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Results of foam stability of pilsner reference beer after addition of hop pectin derived from bines, cones or waste of four different hop varieties.

Samples	g pectin preparation/hi	Pectin addition as AUA g AUA/hi	Foam stability (sec)	Foam improvement (sec)
<b>Hersbrucker</b>				
bine	5	3.3	348	42
	10	6.6	354	50
cone	5	3.05	351	47
	10	6.1	365	61
waste	5	2.75	-	-
	10	5.5	345	41
<b>Aroma perle</b>				
bine	5	3.75	343	39
	10	7.5	374	70
cone	5	3.25	342	38
	10	6.5	366	62
waste	5	2.8	339	35
	10	5.6	351	47
<b>Northern brewer</b>				
cone	5	2.8	346	42
	10	5.6	360	56
waste	5	3.6	334	30
	10	7.2	360	56
<b>Brewers gold</b>				
bine	5	3.2	336	32
	10	6.4	368	64
cone	5	3.0	341	37
	10	6.0	359	55
waste	5	3.25	343	39
	10	6.5	352	48
Reference	-	-	304	-

4. At that time the results of these experiments were surprising and unexpected. It was surprising and unexpected that the addition of hop pectin would improve the foam stability of reference beer in the dramatic fashion shown by the test results above.

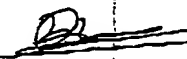
22.NOV.1999 15:36

HEINEKEN HTS R&amp;D

5. The undersigned declares that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that wilful false statements so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such wilful false statements so made may jeopardize the validity of the document, or application, or any patent issuing thereon.

Signed this 22<sup>nd</sup> day of November, 1999

By

  
Albert Doderer

OFFICIAL